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WITNESS my hand this  
Twenty-ninth day of October 2004

A handwritten signature in black ink, appearing to be 'L. Mynott'.

LEANNE MYNOTT  
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AUSTRALIA

Patents Act 1990

**PROVISIONAL SPECIFICATION**

**Applicant:**

BHP BILLITON INNOVATION PTY LTD

A.C.N. 008 457 157

**Invention Title:**

ELECTROCHEMICAL REDUCTION OF METAL OXIDES

The invention is described in the following statement:

## ELECTROCHEMICAL REDUCTION OF METAL OXIDES

The present invention relates to electrochemical reduction of metal oxides.

5

The present invention relates particularly to continuous and semi-continuous electrochemical reduction of metal oxides in the form of powder to produce metal having a low oxygen concentration, typically no more than 10 0.2% by weight.

The present invention was made during the course of an on-going research project on electrochemical reduction of metal oxides being carried out by the 15 applicant. The research project has focussed on the reduction of titania ( $\text{TiO}_2$ ).

During the course of the research project the applicant carried out experimental work on the reduction 20 of titania using electrolytic cells that included a pool of molten  $\text{CaCl}_2$ -based electrolyte, an anode formed from graphite, and a range of cathodes.

The  $\text{CaCl}_2$ -based electrolyte was a commercially 25 available source of  $\text{CaCl}_2$ , namely calcium chloride dihydrate, that decomposed on heating and produced a very small amount of  $\text{CaO}$ .

The applicant operated the electrolytic cells at 30 a potential above the decomposition potential of  $\text{CaO}$  and below the decomposition potential of  $\text{CaCl}_2$ .

The applicant found that at these potentials the cell could electrochemically reduce titania to titanium 35 with low concentrations of oxygen, ie concentrations less than 0.2 wt.%. .

The applicant operated the electrolytic cells on a batch basis with titania in the form of pellets and larger solid blocks in the early part of the work and titania powder in the later part of the work. The  
5 applicant also operated the electrolytic cells on a batch basis with other metal oxides.

Whilst the research work established that it is possible to electrochemically reduce titania (and other  
10 metal oxides) to metals having low concentrations of oxygen in such electrolytic cells, the applicant has realised that there are significant practical difficulties operating the electrolytic cells commercially on a batch basis.

15 In the course of considering the results of the research work and possible commercialisation of the technology, the applicant realised that it was possible that commercial production could be achieved by operating  
20 an electrolytic cell on a continuous or semi-continuous basis with metal oxide powders and pellets being transported through the cell in a controlled manner and being discharged in a reduced form from the cell.

25 Australian provisional application 2002953282 lodged on 12 December 2002 in the name of the applicant describes this invention in broad terms as a process for electrochemically reducing a metal oxide, such as titania, in a solid state in an electrolytic cell that includes a  
30 bath of molten electrolyte, a cathode, and an anode, which process includes the steps of: (a) applying a cell potential across the anode and the cathode that is capable of electrochemically reducing metal oxide supplied to the bath, (b) continuously or semi-continuously feeding the  
35 metal oxide in powder form into the bath, (c) transporting the powder along a path within the bath and reducing the metal oxide to metal as the metal oxide powder moves along

the path, and (d) continuously or semi-continuously removing metal from the bath.

5       The Australian provisional application defines the term "powder" as meaning particles having a particle size of 3.5 mm or less. This particle size range covers particles at the upper end of the size range that can also be described as pellets.

10       The term "semi-continuously" is understood in the Australian provisional application and herein to mean that the process includes: (a) periods during which metal oxide powder and pellets are supplied to the cell and periods during which there is no such supply of metal oxide powder  
15       and pellets to the cell, and (b) periods during which metal is removed from the cell and periods during which there is no such removal of metal from the cell.

20       The overall intention of the use of the terms "continuously" and "semi-continuously" in the Australian provisional application and herein is to describe cell operation other than on a batch basis.

25       In this context, the term "batch" is understood in the Australian provisional application and herein to include situations in which metal oxide is continuously supplied to a cell and reduced metal builds up in the cell until the end of a cell cycle, such as disclosed in International application WO 01/62996 in the name of The  
30       Secretary of State for Defence.

35       After making the initial invention described above, the applicant carried out further research into the possibility of commercial production based on operating an electrolytic cell on a continuous or semi-continuous basis. The applicant realised that a commercial production cell should include a cell cathode in the form

of a member, such as a plate, having an upper surface for supporting metal oxides in pellet form, as described herein, that is horizontally disposed or slightly inclined and has a forward end and a rearward end and is immersed  
5 in the electrolyte bath and is supported for movement, preferably in forward and rearward directions, so as to cause metal oxide pellets to move toward the forward end of the cathode.

10           Australian provisional application 2003903150 lodged on 20 June 2003 in the name of the applicant describes this so-called "shaker table" cathode invention in broad terms.

15           The applicant has carried out further research and development work on the "shaker table" invention and has now designed a particular electrolytic cell in accordance with the invention. The invention of the particular electrolytic cell design is the subject of this  
20 Australian provisional application 2003905261 lodged on 26 September 2003 in the name of the applicant. The particular electrolytic cell design is characterised by multiple anodes and by support structures that separately support the "shaker table" cathode and the anodes from  
25 above the cell, with the support structure enabling adjustment of the spacing of the anodes above the upper surface of the "shaker table" cathode.

30           In the course of the research and development work on the possibility of commercial production based on operating an electrolytic cell on a continuous or semi-continuous basis, the applicant realised that it is important for efficient operation of the cell that there be a continuous or a periodic purge of electrolyte from  
35 the bath over and above that required as make-up for electrolyte that is discharged from the bath with reduced metal and that there be deliberate addition of electrolyte

to make up for the purge.

In the course of the research and development work the applicant also realised that the purged  
5 electrolyte could be cleaned to remove contaminants (such as carbides and carbonates) and that the cleaned electrolyte could be returned to the bath.

According to the present invention there is  
10 provided a process for electrochemically reducing metal oxide feed material in a solid state in an electrolytic cell of the type that includes a molten bath of electrolyte, an anode, a cathode, and a means for applying a potential across the anode and the cathode, which  
15 process includes the steps of: (a) applying a potential across the anode and the cathode that is capable of electrochemically reducing metal oxide supplied to the molten electrolyte bath, (b) continuously or semi-continuously supplying the metal oxide into the bath, (c)  
20 transporting the metal oxide feed material along a path within the bath and reducing the metal oxide to metal as the feed material moves along the path, (d) continuously or semi-continuously removing metal from the bath, (e) supplying an amount of electrolyte into the bath that is  
25 greater than the amount of electrolyte that is required to compensate for loss of electrolyte with metal removed from the bath, and (f) removing molten electrolyte from the bath to maintain the bath height at a required height or within a range of heights.

30

The process above-described process is characterised by step (e) of making a deliberate addition or additions of electrolyte (typically  $\text{CaCl}_2$ ) over and above the amount of electrolyte that is required to make  
35 up for electrolyte that is retained by metal removed from the bath during operation of the cell.

The electrolyte addition in step (e) may be on a continuous or a periodic basis.

5 The electrolyte added to the bath in step (e) may be in a molten phase or a solid phase.

10 Preferably step (e) includes feeding electrolyte in an amount that is between 70% and 100% of the amount of metal oxide feed material supplied to the bath in step (b) on a time averaged basis.

15 The process is also characterised by step (f) of deliberately removing electrolyte from the bath to maintain bath height at the required height or within the range of heights.

In one, although not the only, embodiment, electrolyte removal is via an overflow weir in the cell. The removed electrolyte is treated to remove contaminants, such as carbides and carbonates. The treated electrolyte is returned to the cell.

25 Preferably the metal oxide feed material is in the form of pellets, as described herein.

30 Preferably the process includes treating the electrolyte removed from the bath in step (f) to remove contaminants and feeding the treated electrolyte to the bath.

35 Preferably the cathode is in the form of a member, such as a plate, having an upper surface for supporting metal oxides that are in pellet form that is horizontally disposed or slightly inclined and has a forward end and a rearward end and is immersed in the electrolyte bath and is supported for movement in forward and rearward directions so as to cause metal oxide pellets

to move toward the forward end of the cathode.

5 With this arrangement, preferably step (b) includes supplying the metal oxide feed material in pellet form to the bath so that the pellets deposit on the upper surface of the cathode at a rearward end of the cathode.

10 Preferably the process includes the step of causing metal oxide pellets to move over the upper surface of the cathode toward the forward end of the cathode while in contact with molten electrolyte whereby electrochemical reduction of the metal oxide to metal occurs as the pellets move toward the forward end.

15 Preferably step (b) includes supplying metal oxide pellets into the molten electrolyte bath so that the pellets form a mono-layer on an upper surface of the cathode.

20 Preferably step (c) includes transporting the metal oxide pellets by causing metal oxide pellets to move on the upper surface of the cathode toward the forward end of the cathode as a packed mono-layer layer of pellets.

25 Preferably step (c) includes selectively moving the cathode so as to cause metal oxide pellets on the upper surface of the cathode to move toward the forward end of the cathode.

30 Preferably step (c) includes transporting the metal oxide pellets by moving the cathode so as to cause pellets across the width of the cathode to move at the same rate so that the pellets have substantially the same residence time within the bath.

35 Preferably the process electrochemically reduces the metal oxide to metal having a concentration of oxygen

that is no more than 0.3% by weight.

More preferably the concentration of oxygen is no more than 0.2% by weight.

5

The process may be a single or multiple stage process involving one or more than one electrolytic cell.

10 In a situation in which the metal oxide feed material is in pellet form, preferably the process includes washing pellets that are removed from the bath in step (d) to separate electrolyte that is carried from the cell with the pellets.

15 The process includes recovering electrolyte that is washed from the pellets and recycling the electrolyte to the cell.

20 Preferably the process includes maintaining the cell temperature below the vaporisation and/or decomposition temperatures of the electrolyte.

25 Preferably the process includes applying a cell potential above a decomposition potential of at least one constituent of the electrolyte so that there are cations of a metal other than that of the cathode metal oxide in the electrolyte.

30 In a situation in which the metal oxide is titania it is preferred that the electrolyte be a  $\text{CaCl}_2$ -based electrolyte that includes  $\text{CaO}$  as one of the constituents.

35 In such a situation it is preferred that the process includes maintaining the cell potential above the decomposition potential for  $\text{CaO}$ .

According to the present invention there is provided an electrolytic cell for electrochemically reducing metal oxide feed material which includes (a) a bath of a molten electrolyte, (b) a cathode, (c) an anode, 5 (d) a means for applying a potential across the anodes and the cathode, (e) a means for supplying metal oxide feed material to the electrolyte bath, (f) a means for removing at least partially electrochemically reduced metal oxides from the electrolyte bath, (g) a means for supplying an 10 amount of electrolyte into the bath that is greater than the amount of electrolyte that is required to compensate for loss of electrolyte with metal removed from the bath, and (f) a means for removing molten electrolyte from the bath to maintain the bath height at a required height or 15 within a range of heights.

Preferably the cell further includes a means for treating the electrolyte removed from the bath in step (f) to remove contaminants from the electrolyte and for 20 feeding the treated electrolyte to the bath. As indicated above, the targeted contaminants may include carbides and carbonates.

Preferably the means for applying a potential 25 across the anodes and the cathode includes (a) a power source and (b) an electrical circuit that electrically interconnects the power source, the anodes, and the cathode.

30 Preferably the cell includes a means for treating gases released from the cell.

The gas treatment means may include a means for removing any one or more of carbon monoxide, carbon 35 dioxide, chlorine-containing gases, and phosgene from the gases.

The gas treatment means may also include a means for combusting carbon monoxide gas in the gases.

5 In a situation in which the metal oxide is titania it is preferred that the electrolyte be a  $\text{CaCl}_2$ -based electrolyte that includes  $\text{CaO}$  as one of the constituents.

10 Preferably the particle size of the pellets is in the range of 1-4 mm.

Typically, the particle size of the pellets is in the range of 1-3 mm.

15 The present invention is described further by way of example with reference to the accompanying drawings of which:

20 Figure 1 is a schematic diagram that illustrates one embodiment of an electrochemical process and apparatus in accordance with the present invention;

25 Figure 2 is a perspective view of the electrolytic cell of the apparatus shown in Figure 1, with the cathode support rods removed to clarify the Figure;

Figure 3 is a vertical section through the electrolytic cell shown in Figures 1 and 2;

30 Figure 4 is the vertical section shown in Figure 3 with the top cover and the anodes and the anode support structure removed to illustrate the cathode and the cathode support structure more clearly; and

35 Figure 5 is the vertical section shown in Figure 3 with the top cover and the cathode and the cathode support structure removed to illustrate the anodes and the

anode support structure more clearly.

The following description is in the context of electrochemically reducing titania pellets to titanium metal having an oxygen concentration of less than 0.3 wt.%. However, it is noted that the present invention is not confined to this metal oxide and extends to other metal oxides in pellet or other form.

The process is characterised by a step of making deliberate addition of electrolyte (typically  $\text{CaCl}_2$ ) over and above the amount of electrolyte that is required to make up for electrolyte that is retained by metal removed from the bath during operation of the cell.

The process is also characterised by a step of deliberately removing electrolyte from the bath to maintain bath height within the cell.

In the subject embodiment, electrolyte addition is in the form of suitably-sized cast blocks and electrolyte removal is via an overflow weir in the cell. The removed electrolyte is treated to remove  $\text{CaO}$  (by washing with  $\text{HCl}$ ) and optionally contaminants, such as carbon. The treated electrolyte is returned to the cell. In effect, removal of electrolyte and treatment of the electrolyte removes oxygen ions from the electrolyte. This is a benefit in terms of improving the rate of diffusion of oxygen ions from the cathode to the anode.

The current proposal is to add 0.8kg/hr titania and 1.0 kg/hr electrolyte to the cell. This is a relatively small addition of electrolyte in the context that the bath will hold approximately 700 kg salt but is a relatively substantial addition in the context of the addition rate for titania to the cell.

The main component of the apparatus shown in Figure 1 is an electrolytic cell 1.

5 The cell shown in the drawings is an enclosed chamber, although not completely sealed, that is rectangular in top plan and has a base wall 3, a pair of opposed end walls 5, a pair of opposed side walls 7, and a top cover 9.

10 The cell contains a bath 21 of molten electrolyte. The preferred electrolyte is  $\text{CaCl}_2$  with at least some  $\text{CaO}$ .

15 The cell includes an inlet 59 (see Figure 2) for solid electrolyte blocks in the top cover 9 at the right hand end as viewed in Figure 2 and the left hand end as viewed in the other Figures.

20 The apparatus includes a furnace 45 for melting commercially-supplied electrolyte and a casting station 47 for casting molten electrolyte from the furnace into suitably-sized blocks.

25 The cell includes a series of inlets for titania pellets in the top cover 9 near the left hand end of the cell as viewed in Figures 1, 3, 4, and 5 and near the right hand end of the cell as viewed in Figure 2. This end of the cell is hereinafter referred to as "the rearward end" of the cell. The inlets are identified by the numeral 11  
30 in Figure 2.

The apparatus includes a pan pelletiser 51 that forms the pellets in a "green" state and a sintering furnace 53 that sinters the "green" pellets to a  
35 sufficient strength to withstand subsequent processing. The sintered pellets are stored in a storage bin 55 ready to be supplied via a vibratory feeder 57 to the cell

inlets 11. Typically, the pellets have a size range of 1-4 mm.

5 The cell further includes an outlet in the form of an overflow weir 49 (see Figures 3, 4, and 5) for removing excess electrolyte from the cell 1. The overflow weir 49 is located at the opposite end of the cell 1 to the electrolyte inlet 59. The overflow weir 49 is an effective option for ensuring that the bath of electrolyte  
10 does not exceed a predetermined maximum height.

The apparatus also includes a tank 57 for electrolyte removed from the cell 1 via the overflow weir 49.

15

The apparatus also includes a treatment station (not shown) for treating the electrolyte from tank 57 prior to recycling the electrolyte to the cell 1. For example, the electrolyte may be treated to remove carbides  
20 and carbonates from the electrolyte. Treatment may also include otherwise treating the electrolyte to remove other contaminants.

The cell further includes an outlet 13 for  
25 titanium metal pellets in the base wall 3 near the right hand end of the cell as viewed in Figures 1, 3, 4, and 5 and near the right hand end of the cell as viewed in Figure 2. This end of the cell is hereinafter referred to as "the forward end" of the cell. The outlet 13 is in the form of  
30 a sump defined by downwardly converging sides 15 and an upwardly inclined auger 35 or other suitable means arranged to receive titanium pellets and retained electrolyte from a lower end of the sump and to transport the pellets away from the cell.

35

The cell further includes a cathode 25 in the form of a plate or other suitable member that is immersed

in the bath 21 and is positioned a short distance above the base wall 3. The cathode plate 25 is supported in the cell by a support structure so that the upper surface of the cathode plate 25 is horizontal or slightly inclined downwardly from the rearward end to the forward end of the cell. The length and width dimensions of the cathode plate 25 are selected to be as large as possible to fit conveniently within the cell. The cathode plate 25 is supported to move in the forward and rearward directions in an oscillating motion as described hereinafter. The cathode support structure is described in detail in Australian provisional application 2003905261 in the name of the applicant and the disclosure in the provisional application is incorporated herein by cross-reference.

15

The cell further includes 6 anodes generally identified by the numeral 19 that extend into the bath 21. The anodes 19 include graphite blocks 23 mounted to the ends of rods or other suitable support members 27. The anode blocks 19 include lengthwise extending slots 91 (see Figure 2) that allow gas that evolves in the electrolyte bath 21 to escape from the cell. The anodes 19 are arranged in pairs and the size of the anode blocks 23 is selected so that the anodes are positioned directly above substantially the whole of the upper surface of the cathode plate 25. The anodes 19 are supported by a support structure so that the anode blocks 23 can be progressively lowered into the bath 21 as lower sections of the anode graphite are consumed by cell reactions at the anodes. The top cover 9 of the cell includes openings 95 (see Figures 2 and 3) for the support members 27. The anode support structure is described in detail in the above-mentioned Australian provisional application 2003905261.

35

The applicant has found that movement of the cathode plate 25 in a repeated sequence that comprises a

short period of forward and backward, ie oscillating, motion and a short rest period can cause pellets on the upper surface of the cathode plate 25 to move over the upper surface in a series of short steps from the rearward end to the forward end of the cell.

Moreover, the applicant has found that the above-described type of motion can cause pellets across the width of the cathode plate 25 to move at a constant rate so that the pellets have substantially the same residence time within the bath 21.

The cell further includes a power source 31 for applying a potential across the anode block 23 and the cathode plate 25 and an electrical circuit (that includes the above-described cathode support members 79) electrically interconnects the power source 31, the anodes 23, and the cathode. The size and/or the positions of the cathode support members 79 is selected to supply a preselected current distribution to the cathode plate 25 to optimise electrochemical reduction of titania pellets on the cathode plate 25. Depending on the circumstances, there may be a range of current distributions required in the operation of the cell.

The operation of the cell generates carbon monoxide and carbon dioxide and potentially chlorine-containing gases at the anode blocks 23 and it is important to remove these gases from the cell. The cell further includes an off-gas outlet 41 in the top cover 9 of the cell and a gas treatment unit 43 that treats the off-gases before releasing the treated gases to atmosphere. The gas treatment includes removing carbon dioxide and any chlorine gases and may also include combustion of carbon monoxide to generate heat for the process.

In use of the cell, titania pellets are supplied to the upper surface of the cathode plate 25 at the rearward end of the cell so as to form a mono-layer of pellets on the cathode plate 25 and the cathode is moved  
5 as described above and causes the pellets to step forward over the surface of the plate to the forward end of the cell and ultimately fall from the forward end of the cathode. The pellets are progressively electrochemically reduced in the cell as the pellets are moved over the  
10 surface of the cathode plate 25. The operating parameters of the cathode plate 25 are selected so that the pellets have sufficient residence time in the cell to achieve a required level of reduction of the titania pellets. Typically, 2-4 mm titania pellets require 4 hours  
15 residence time to be reduced to titanium with a concentration of 0.3 wt% oxygen at a cell operating voltage of 3 V.

The applicant has found that the above-described  
20 arrangement results in substantial reduction of titania pellets within a short distance from the forward end of the cell.

Titanium pellets, together with electrolyte that  
25 is retained in the pores of the titanium pellets, are removed from the cell continuously or semi-continuously at the outlet 13. The discharged material is transported via the auger 35 to a water spray chamber 37 and quenched to a temperature that is below the solidification temperature  
30 of the electrolyte, whereby the electrolyte blocks direct exposure of the metal and thereby restricts oxidation of the metal. The discharged material is then washed to separate the retained electrolyte from the metal powder. The metal powder is thereafter processed as required to  
35 produce end products.

Electrolyte in amounts greater than that required

to compensate for net loss of electrolyte with discharged titanium pellets is added to the cell in the form of cast electrolyte blocks via inlet 59. As is indicated above, the purpose of the additional electrolyte is to purge the cell to maintain optimum operating conditions. As electrolyte is added to the cell via the inlet 59 at the rearward end of the cell, electrolyte flows over the weir 49 at the forward end of the cell and flows into the storage tank 57 and thereafter is treated by being washed with HCl to remove CaO and the treated electrolyte is returned to the cell as part of the electrolyte addition to the cell.

The anode blocks 23 are progressively consumed during operation of the cell by a reaction between carbon in the anode block 23 and  $O^{--}$  anions generated at the cathode plate 25, and the reaction occurs predominantly at the lower edges of the anode blocks 23. The distances between the upper surface of the cathode plate 25 and the lower edges of the anode blocks 23 are maintained as required to maintain optimum operating conditions in the cell. Preferably the distance between the upper surface of the cathode plate 25 and the lower edges of the anode block 23 is selected so that there is sufficient resistance heating generated to maintain the bath 21 at a required operating temperature.

Preferably the cell is operated at a potential that is above the decomposition potential of CaO in the electrolytic. Depending on the circumstances, the potential may be as high as 4-5V. In accordance with the above-described mechanism, operating above the decomposition potential of CaO facilitates deposition of Ca metal on the cathode plate 25 due to the presence of  $Ca^{++}$  cations and migration of  $O^{--}$  anions to the anode block 23 as a consequence of the applied field and reaction of the  $O^{--}$  anions with carbon of the anode block 23 to

generate carbon monoxide and carbon dioxide and release electrons. In addition, in accordance with the above-described mechanism, the deposition of Ca metal results in chemical reduction of titania via the mechanism described above and generates  $O^{2-}$  anions that migrate to the anode block 23 as a consequence of the applied field and further release of electrons. Operating the cell below the decomposition potential of  $CaCl_2$  minimises evolution of chlorine gas, and is an advantage on this basis.

10

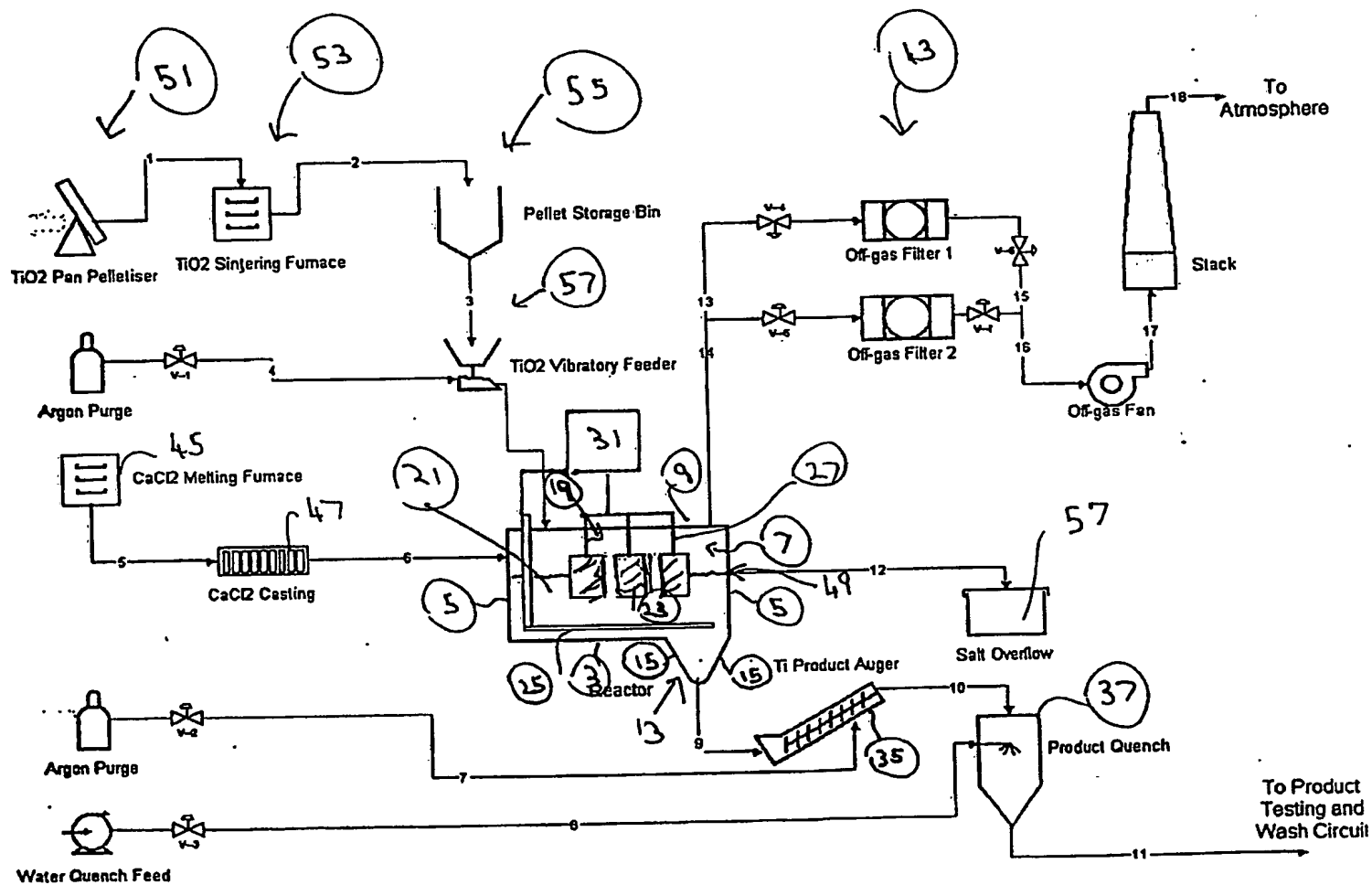
The above-described cell and process are an efficient and an effective means of continuously and semi-continuously electrochemically reducing metal oxides in the form of pellets to produce metal having a low oxygen concentration

15

Specifically, the electrolytic cell shown in the drawing is one example only of a large number of possible cell configurations that are within the scope of the present invention.

20

Figure 1



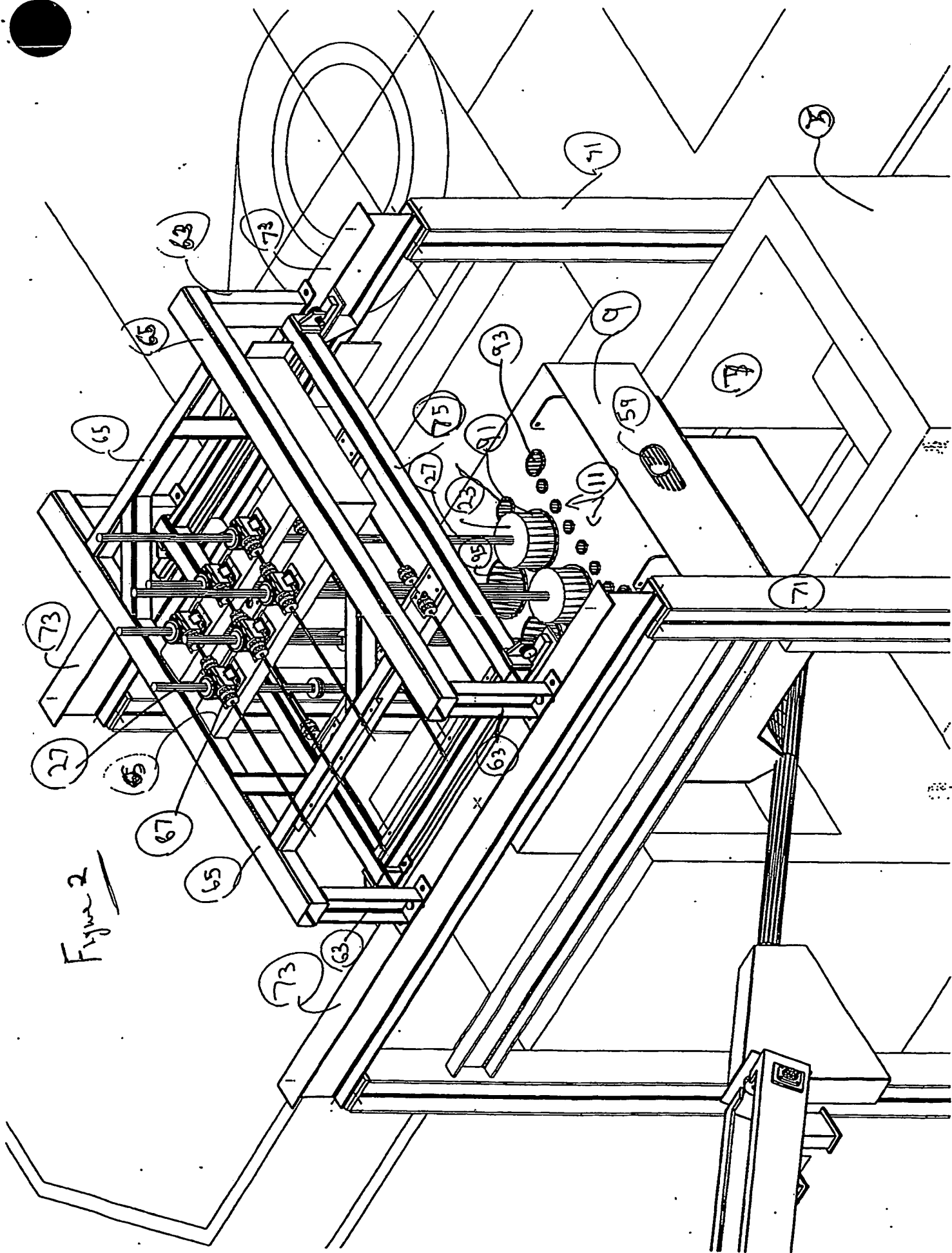


Figure 2

Figure 3

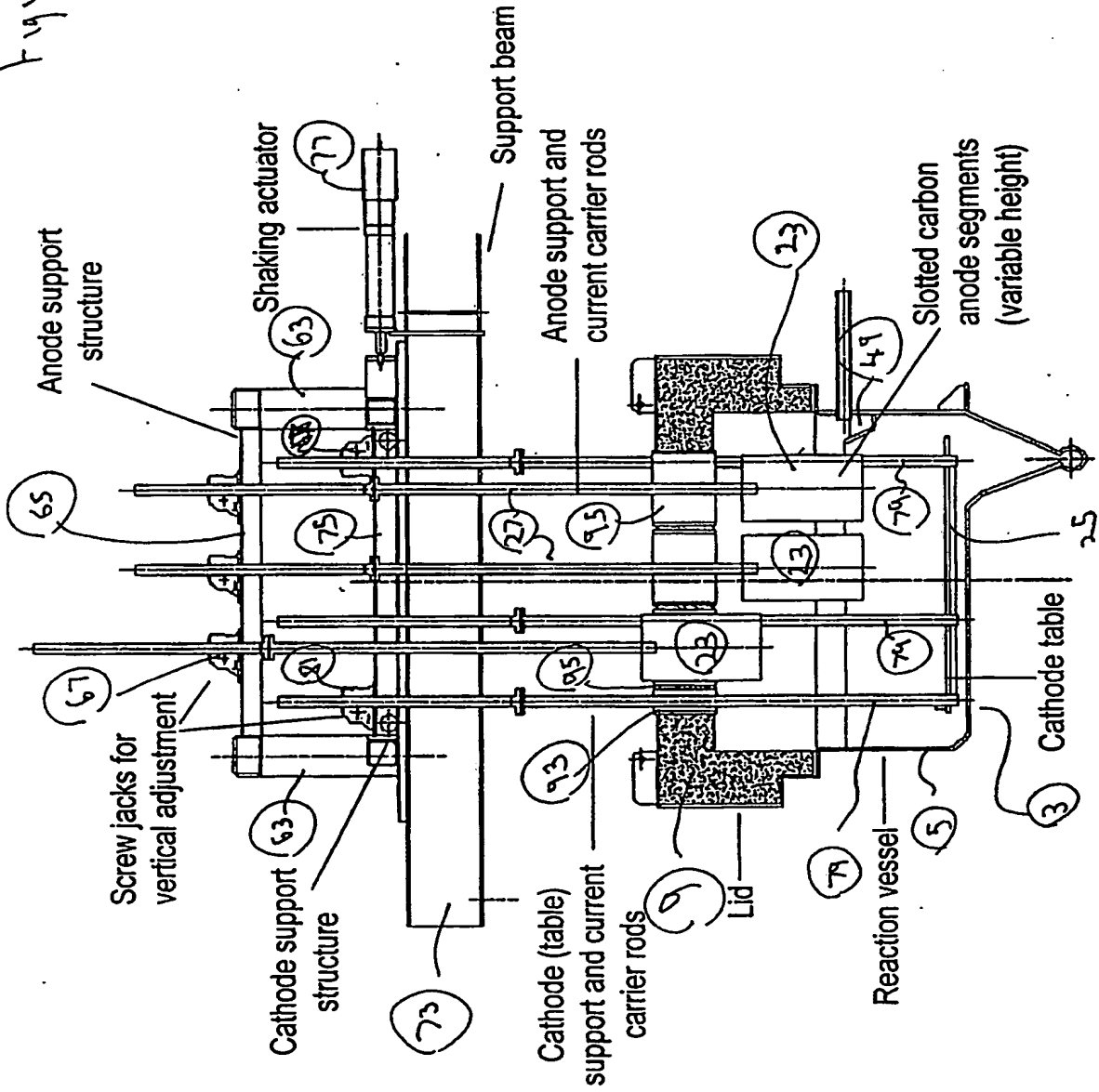
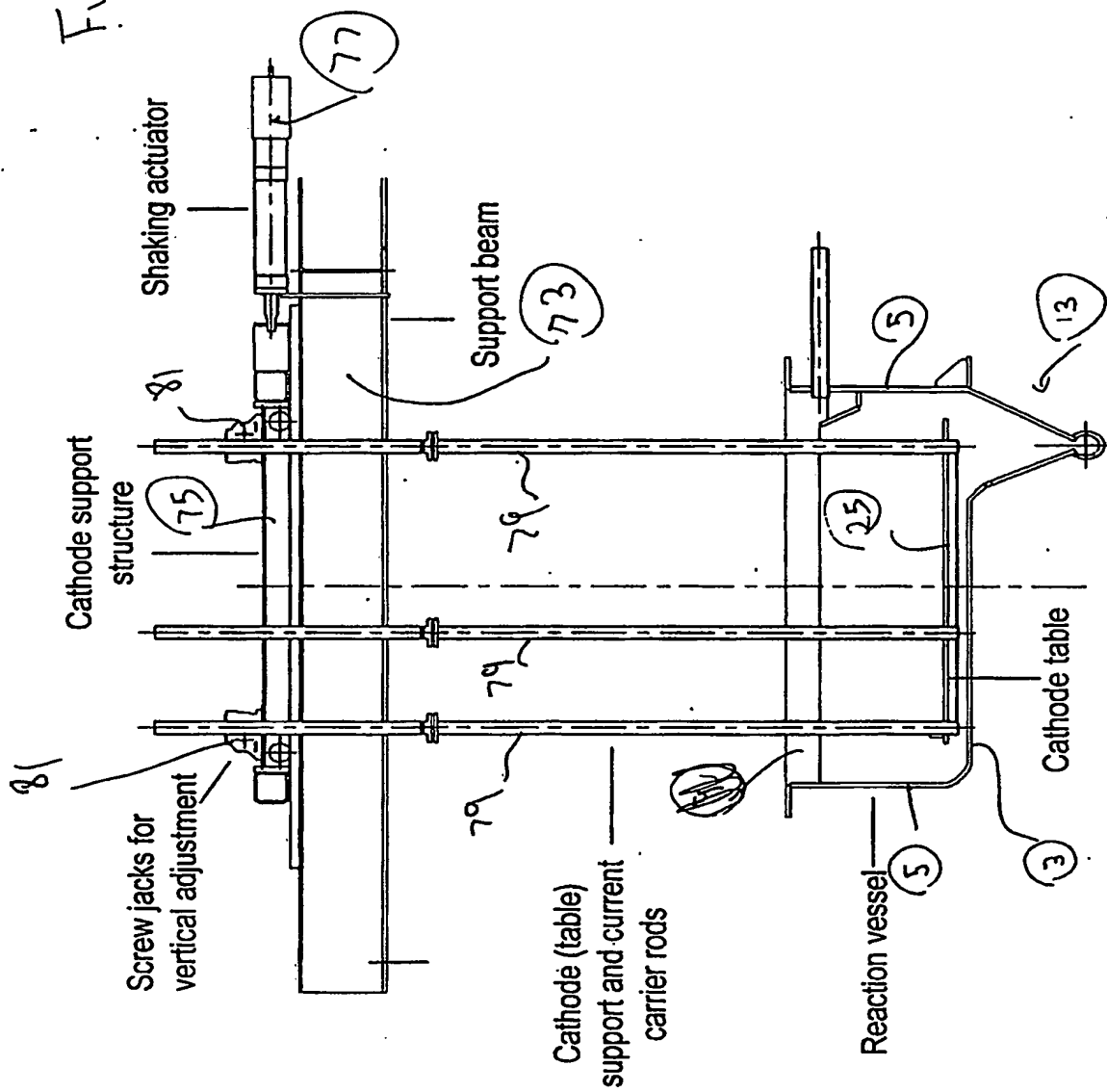


Figure 4





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